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PROCEDURE AND CRITERIA FOR EVALUATING NEW INSTRUMENTAL MEASUREMENT METHOD PRINCIPLES (NIMMP) FOR THE MUNICIPAL AND INDUSTRIAL STRATEGY FOR ABATEMENT (MISA) PROGRAM

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Environment Ontario

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Ontario Ministry of the Environment

Protocol and Criteria for Evaluating New Instrumental Measurement Method Principles (NIMMP)

Introduction

This protocol describes the procedure for the submission of proposals for New Instrumental Measurement Method Principles (NIMMP) for the MISA program. NIMMP refer to analytical principles not included in, or which differ from the approved Instrumental Measurement Method Principles (IMMP), and Alternate Instrumental Method Principles, listed in columns 4 and 5 of Schedule 3 Part A, and Schedule 3 Part B of Ontario Regulation 695/88 as amended to Ontario Regulation 533/89 under the Environmental Protection Act, Effluent Monitoring - General, or listed in equivalent schedules of the Monitoring Regulations for specific Sectors. These regulations are called "MISA Regulations" in this protocol.

The Ontario Ministry of the Environment (MOE) specifies IMMP, and Alternates, in its MISA Regulations. There are no detailed method procedures in the schedules of the MISA Regulations. The principles listed in the Regulations were chosen because they were widely applicable and could be expected to serve as the basis for the development of reliable methods. The MOE encourages and supports the search for NIMMP which may reduce analytical cost, or provide more reliable or more comprehensive data.

Proposals for NIMMP follow two stages: preliminary and detailed. If a detailed proposal is required, the proponent must apply method procedures based on one of the approved IMMP, as a benchmark, to show that the NIMMP perform in an equivalent or superior manner.

Terms of Reference

New instrumental method measurement principles (NIMMP) will be considered only under the following conditions:

1) The NIMMP may be used for MISA only subsequent to the Effluent Monitoring Regulations.

2) The NIMMP must be supported by more than half of the members on the Joint Technical Committee (JTC) for a MISA Sector.

3) The NIMMP must be capable of achieving the method detection limit (MDL) as specified in column 6 of Schedule 3 Part A, and Schedule 3 Part B, Ontario Regulation 695/88 under the Environmental Protection Act, Effluent Monitoring - General, or listed in equivalent schedules in the Monitoring Regulations for specific Sectors, using the procedures outlined in the publication "Estimation of Analytical Method Detection Limits (MDL)", Ontario Ministry of the Environment, Queen's Printer for Ontario, 1988, ISBN 0-7729-4117-3.

Proposal Procedure

The NIMMP must be proposed by a member of the JTC for a MISA Sector; this person is called the "proponent". When a decision is made to approve or reject the NIMMP, the proponent and MOE Sector representatives on all other MISA JTC will be notified in writing by the MOE Sector representative, giving reasons for the decision. The steps leading to evaluation of the NIMMP follow:

1) The proponent assembles a brief information package which outlines the instrumentation and technology involved in the analysis, technical references or publications, the advantage(s) of the NIMMP, sample preparation, analytical procedures, known interferences, and the calculation of the MDL. The package should be provided to all MISA JTC members at least two weeks in advance of a meeting which has the NIMMP on the agenda.

2) Discussion of the NIMMP at the MISA JTC meeting will be limited to rejecting the NIMMP, or approving the NIMMP for further consideration.

    a) If the NIMMP proposal is rejected, the reason(s) will be noted.

    b) To allow further consideration, the NIMMP must be supported by more than half of the JTC members. Absent members may vote verbally or in writing, prior to the meeting.

3) If the NIMMP proposal is approved for further consideration, the MOE representative(s) of the JTC will report back to the JTC within three weeks: the NIMMP may be approved, or require a full-scale evaluation.

    a) NIMMP that are well-supported or established may be approved for inclusion in future MISA Regulations (Sector-specific) without further documentation.

    b) If a full-scale evaluation is required, the proponent must supply detailed documentation and data, as described below, to the representative(s) of the MOE on the JTC. The MOE representative(s) will report back to the JTC within six weeks: the NIMMP may be approved, rejected, or further information requested. If the proposal is still incomplete after resubmission, it will be rejected. Rejected NIMMP will not be reconsidered for a Sector for one year from the date of rejection. Approved or rejected NIMMP may be proposed for other MISA Sectors, but extra documentation must be supplied as described below.

Documentation Required

The detailed proposal must contain at least the following documents for both the benchmark method procedures using approved IMMP, and method procedures using the NIMMP:

1) Applicable literature citations or other references;

2) Instrument specifications. Instrument specifications shall include the measurement principle and a description of: all major components of the analytical system including, if appropriate, the sampler or injection port, pumps, valves, fittings, materials contacting the sample, column(s), gas(es), analytical range(s), power and other support requirements, temperature operating range, detector sensitivity, output readability, and repeatability.

3) Complete analytical procedures. The procedures shall contain a description of: the sample matrix for which the NIMMP are intended, potential safety hazards, quantity of waste produced and characteristics, potential interferences and limitations of the instrument and procedure, instrumentation/equipment, manifold/flow diagrams, reagents, calibration and QC standards, quality control procedures and control limits, sample preservation, sample preparation including dilution or preconcentration, sample spiking procedures, instrument operation, the analytical procedure, QC and sample run order, calculation of final results, QA procedures, advantage(s) of NIMMP and associated method procedures.

4) The range of concentrations of the parameter(s) found in effluents from all sampling points in the Sector with method procedures using approved IMMP, along with the highest concentrations of potential interferences expected in these effluents.

5) A list of potential sources of determinate error or interferences along with procedures to minimize or quantify the error/interference, and quality control protocols to ensure that these procedures work effectively.

If the NIMMP have been previously approved or rejected for any other Sector(s) the proponent shall supply (where appropriate):

Documents arising from the evaluation of the NIMMP with specific evidence that the reasons for approval also apply to the current Sector, or specific evidence that the reasons for rejection have been overcome or do not apply to the current Sector. The proponent should provide this information as a preface to all other documents.

Data Required

Data are required to demonstrate the performance of the NIMMP. As a minimum, the proponent shall supply the following data, appropriately labelled and summarized (e.g. mean and standard deviation) for both the benchmark method procedures and the method procedures using NIMMP:

1) Calibration graphs with at least five points distributed over the analytical range showing the relationship between concentration, or weight, or pH, and the instrument response for at least eight separate operating days when samples were analyzed;

2) Quality control charts (Shewhart or equivalent) showing QC data and control limits for blanks, spiked blanks, spiked samples, and duplicates, where appropriate, for at least eight separate operating days when samples were analyzed. Both slope control and intercept control of the calibration must be demonstrated.

3) Comparability data from the analysis of samples collected from five effluent streams or waste representative of those regulated under MISA for the Sector, and in which the concentrations range from the method detection limit (MDL) to the maximum contaminant level (MCL) expected for the Sector. From each of these streams or waste, four samples shall be collected and each sample analyzed eight times, four times each by the proposed method procedures using NIMMP and method procedures using approved IMMP for the particular test or test group. Samples may be spiked to provide data spanning the range from the MDL to the MCL, but such data must be clearly indicated. Spiked blanks cannot be substituted for Sector samples. Regression plots and regression statistics must be provided.

4) Data used to calculate the MDL.

5) Results from an interference study. In addition, any interference reported for the procedure in the literature or suspected from method principles must be investigated. The study should show the effect of the interfering substances at the highest concentrations expected in samples from the Sector, and include treatments, if available, to counteract the interference.

6) Results from spiked samples including expected or theoretical result. 25% of the samples collected for the comparability check shall be spiked with the parameter(s) or surrogates from the test group. These data are in addition to any obtained for subsection 3 above using spiked samples.

7) Results from samples which have been diluted a further 50%, where appropriate, to test for matrix effects. Independently, dilute 25% of the samples collected for the comparability check a further 50% with distilled deionized water, and analyze them in the same set as the "undiluted" samples.

8) Results from the analysis of laboratory blank water if appropriate.

9) Results from the analysis of standard reference materials if available.

10) Recovery results. Analyze several forms of the target contaminant -particularly forms expected in effluent (e.g. dissolved organic carbon: analyze ring and straight chain carbon compounds) if appropriate.

11) Additional data where appropriate to demonstrate dynamic range, precision, accuracy, and ruggedness of the method procedures used to propose the NIMMP.

If the NIMMP have been previously approved or rejected for any other Sector(s) the proponent shall supply (where appropriate):

Data showing that the reasons for approval also apply to the current Sector, or that the reasons for rejection have been overcome or do not apply to the current Sector. The proponent should provide this information as a preface to all other documents.

Criteria for Evaluation of Documents and Data

Several criteria will be used to evaluate the information supplied by the proponent:

1) The information must be complete and organized in a logical fashion. All of the points in this protocol must be addressed or the proposal will be returned for further details.

2) Procedures described must be consistent with standards of good laboratory practice identified in the document "Code of Practice for Environmental Laboratories", D. King, Laboratory Services Branch, Ontario Ministry of the Environment, September, Queen's Printer for Ontario, 1989 (ISBN 0-7729-5874-2).

3) The accuracy, precision, and recovery results will be compared to results expected from method procedures using approved IMMP. In general, results should be equivalent or superior to be accepted.

4) Regression statistics will be evaluated with regard to the distribution of points on the line, apparent curvature, or obvious bias. The regression must extend over the range of concentrations representative of the Sector.

5) The data for the benchmark method must show performance that is expected of procedures in the regulations (i.e. the NIMMP will not be approved if compared to a poorly-executed reference method).

6) Literature references will be evaluated on the basis of relevancy to the analysis of MISA effluent or waste samples.

7) Other pertinent details provided by the proponent will be considered: several minor problems in the NIMMP proposal may be equivalent to a major problem.

8) Documents arising from the evaluation of the NIMMP for any other Sector(s) will be used to determine if the reasons for approval also apply to the current Sector, or if the reasons for rejection have been overcome or do not apply to the current Sector. When this criterion applies, it will be used to evaluate proposals before any other criteria will be considered.